

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date  
28 October 2004 (28.10.2004)

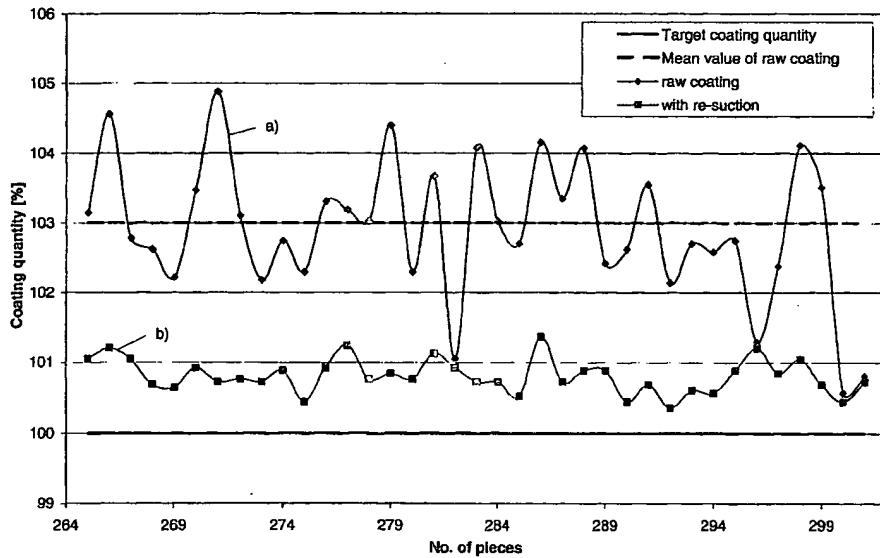
PCT

(10) International Publication Number  
**WO 2004/091786 A1**

- (51) International Patent Classification<sup>7</sup>: **B01J 37/02,**  
35/04, B05D 7/22
- (21) International Application Number:  
PCT/EP2004/004074
- (22) International Filing Date: 16 April 2004 (16.04.2004)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:  
103 17 885.6 17 April 2003 (17.04.2003) DE
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- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

[Continued on next page]

(54) Title: METHOD AND APPARATUS FOR COATING A CARRIER



WO 2004/091786 A1

(57) Abstract: This invention relates to a method and a corresponding apparatus for coating open-pored bodies with at least one coating suspension. In particular, the coating suspension has solids and solutes in a liquid medium in a quantity in wet state which is to correspond to at least a required target quantity. The coating operation has a variation in the applied wet coating quantity from one body to the other. The method according to the invention is characterized by the steps of: coating the body with an actual quantity of the coating suspension, which is always larger than the required target quantity taking the variation of the coating operation into account, determining the difference between the actual quantity and the required target quantity, and reducing the difference between actual quantity and target quantity by removing still wet coating suspension.

**Published:**

- *with international search report*
- *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

2/PCT

10/553359

JC20 Rec'd PCT/PTO 13 OCT 2005

### **Method and apparatus for coating a carrier**

This invention relates to a method for coating a carrier with a coating suspension. More particularly, this invention relates to a method and an apparatus for coating carriers for catalysts, for example automotive exhaust-gas catalysts.

As a rule, the carriers for automotive exhaust-gas catalysts are of a cylindrical shape with two faces and a shell surface, and a large number of flow ducts for the exhaust gases of the internal combustion engines extend from the first face to the second face essentially in parallel to the cylinder axis. These carriers are also referred to as honeycomb carriers.

The cross-sectional shape of the carriers depends on the installation requirements in the motor vehicle. Carriers having a round cross-section, an elliptical or triangular cross-section are widely used. The flow ducts mostly include a square cross-section and are arranged in a closely packed grid pattern over the entire cross-section of the carriers. Depending on the application, the duct or cell density of the flow ducts varies between 10 and 140 cm<sup>-2</sup>. Honeycomb carriers having cell densities of up to 250 cm<sup>-2</sup> are being developed.

For purifying automotive exhaust gases, catalyst carriers obtained by extruding ceramic bodies are mainly used. Alternatively, catalyst carriers made of corrugated and wound metal foils are available. For purifying passenger car exhaust gases, ceramic carriers having cell densities of 62 cm<sup>-2</sup> are still being used predominantly. The cross-sectional dimensions of the flow ducts are 1.27 x 1.27 mm<sup>2</sup> in this case. Wall thicknesses of such carriers range between 0.1 and 0.2 mm.

In order to convert the pollutants contained in automotive exhaust gases, such as carbon monoxide, hydrocarbons and nitrogen oxides, into harmless compounds, very finely divided metals of the platinum group are typically used, the catalytic effect of which may be altered by compounds of non-

noble metals. These catalytically active components must be deposited onto the carriers. However, it is impossible to guarantee the required very fine dispersion of the catalytically active components by depositing these components onto the geometrical surfaces of the carriers. This applies equally to the non-porous metallic and porous ceramic carriers. A sufficiently large surface for the catalytically active components may be provided only by applying a support layer of finely divided (i.e. in powder form), high-surface area materials onto the internal surfaces of the flow ducts. In the following, this operation is referred to as coating of the carrier. Coating the shell surface of the carriers is not desired and should be avoided in order to prevent loss of valuable catalytically active materials.

A suspension of the finely divided, high-surface area materials in a liquid phase, normally water, is used for coating the carriers. As high-surface area support materials for the catalytically active components, typical coating suspensions for catalytic applications include, for example, aluminum oxides, aluminum silicates, zeolites, silicon dioxide, titanium oxide, zirconium oxide and oxygen-storing components on the basis of cerium oxide. These materials constitute the solids content of the coating suspension. In addition, soluble precursors of promoters or catalytically active noble metals of the platinum group in the periodic table may also be added to the coating suspension. The solids concentration of typical coating suspensions ranges between 20 and 65 wt.-% based on the total weight of the suspension. They exhibit densities between 1.1 and 1.8 kg/l.

According to the prior art, various methods for depositing the support layer onto the carriers using the coating suspension or slurry are known. In order to coat the carriers, they may be dipped into the coating suspension or coated by pouring the coating suspension over them. It is also possible to pump or suck the coating suspension into the ducts of the carriers.

In any case, surplus coating material must be removed from the ducts of the carriers by suction or by blowing-off

with compressed air. This will also open ducts which may have become blocked by coating suspension.

After coating, the carrier and the support layer are dried and then calcined in order to solidify the support layer and fix it to the carrier. Subsequently, the catalytically active components are introduced into the coating by impregnation, using mostly aqueous solutions of precursor compounds of the catalytically active components. As an alternative, the catalytically active components may already be added to the coating suspension itself. In this case, subsequent impregnation of the completed support layer with the catalytically active components may be omitted.

An essential criterion of the coating methods is the coating or loading concentration which can be achieved in a single run using these methods. This signifies the solids content left on the carrier following drying and calcination. The coating concentration is indicated in grams per liter of volume of the carriers (g/l). In practice, coating concentrations of up to 300 g/l are needed for automotive exhaust-gas catalysts. If the method used is incapable of applying this quantity in a single run, the coating operation, following drying and, if necessary, calcination of the carrier, must be repeated until the desired loading is achieved. Frequently, two or more coating operations using coating suspensions of different compositions are performed. As a result, catalysts are obtained which include several layers stacked on top of each other and having different catalytic functions.

DE 40 40 150 C2 describes a method in which catalyst carriers having a honeycomb shape may be coated uniformly with a support layer and a catalytically active layer, respectively, over their entire lengths. Below, catalyst carriers will also be referred to as honeycomb carriers. According to the method described in DE 40 40 150 C2, the cylinder axis of the honeycomb carrier is aligned vertically for coating. Then, the coating suspension is pumped into the ducts through the lower face of the honeycomb carrier until it emerges at the upper face. After that, the coating suspension is pumped down again, and surplus coating suspension is blown or sucked

out of the ducts in order to prevent the ducts from becoming blocked. This method produces support layers which exhibit good uniformity over the entire length of the honeycomb carriers.

The coating method described above includes a certain variation in coating quantities from one carrier to the other. This variation depends on the nature of the coating suspension and on the characteristics of the honeycomb carriers to be coated, such as their porosity, for example.

The object of the present invention is to provide an improved method and an improved apparatus for coating carriers, especially carriers for catalysts, with a coating suspension, which enable the variation in coating quantities to be reduced.

This object is solved with the features according to the claims.

Before describing the invention in detail, a number of terms will be explained below.

In the following, the term "bodies" means inert carriers for catalytically active coatings.

As used below, wet uptake or wet coating quantity describes the quantity of coating suspension remaining on the carriers after coating and before a possible drying operation. It may be determined by weighing the carrier before and after coating.

By contrast, dry uptake is the quantity of coating material present on the carriers following drying and calcination.

In the following, target quantity means the wet uptake which is absolutely necessary in order to achieve the required catalytic activity and which no coated carrier must fall short of.

This invention relates to a method and a corresponding apparatus for coating open-pored bodies with at least one coating suspension. The coating suspension includes solids and solutes in a liquid medium. Coating is performed using a quantity in wet state which is to correspond to at least a required target quantity. The coating operation has a varia-

tion in the applied wet coating quantity from one body to the other. The method according to the invention is characterized by the steps of:

a) coating a body with an actual quantity of the coating suspension which is always greater than the required target quantity taking the variation of the coating operation into account,

b) determining the difference between the actual quantity and the required target quantity, and

c) reducing the difference between the actual quantity and the target quantity by removing still wet coating suspension.

The method is suitable for coating carriers made of metal or ceramics. The carriers may be present in the shape of so-called honeycomb carriers having parallel flow ducts open on both sides or may comprise an open-pored foam or fiber structure. However, the method can also be used to coat so-called wall flow filters.

The following explanation of the invention is based on carriers having parallel flow ducts which are employed in large numbers as carriers for catalysts in order to purify the exhaust gases of internal combustion engines.

Coating of the carriers is carried out in a so-called coating station. According to the prior art, a wide variety of examples are known. The description is based on an exemplary coating station, such as that described in the publications DE 40 40 150 A1, EP 0941763 A1, EP 1136462 A1 and EP 1273344 A1, for example.

For coating, the carriers are placed on the coating station and filled with the coating suspension from below by means of a pump or from a pressurized reservoir. After that, surplus coating suspension is either pumped out of the carrier or sucked out by applying a vacuum. Flow ducts which may have become blocked by coating suspension may then be blown free using compressed air, for example.

The resulting coating of the carrier will be referred to as raw coating below. The coating quantity of the raw coating depends on the solids concentration of the coating suspen-

sion, its viscosity and the coating conditions, especially the conditions when removing the surplus coating suspension from the flow ducts of the carriers. Persons skilled in the art are familiar with these relationships and can define the mean value of the actual coating quantity taking the variation of this coating process into account so that no carrier will fall short of the target quantity.

The variation of this conventional coating process depends on the type of coating suspension and other parameters of the coating process. Usually, the variation is  $\pm 5\%$  to  $\pm 10\%$ . Favorably, it may be reduced down to  $\pm 2\%$ .

In order to decrease the variation of this conventional coating process, the invention provides for a re-suction operation of the still wet coating suspension from one end of the carrier so as to match the actual coating quantity with the desired target quantity. Here, intensity and/or duration of re-suction are adjusted in accordance with the determined differential quantity or surplus. Instead of re-suction also blowing with compressed air can be used to adjust the remaining coating quantity on the carrier.

Adjustment of intensity and/or duration of re-suction may, for example, be done by selecting the corresponding values from tables of values for the measured actual quantity which were established in preliminary tests. Alternatively, intensity and/or duration of re-suction may be controlled in accordance with the values for the actual quantity determined on the bodies coated immediately before, intensity and/or duration of re-suction and the thus achieved reduction in the difference between actual and target quantities, that is, depending on incoming weight or deviation from the required target quantity, re-suction is adjusted in a predictive manner such that the actual quantity approximates the target weight or target coating quantity in the carrier as far as possible.

Following re-suction, the coating quantity is preferably determined again by weighing, and re-suction is repeated until the actual coating quantity is within specifications.

Thus, according to the invention, a conventional coating method is used to apply a raw coating onto the carrier. This is followed by re-suction during which any surplus coating suspension (based on a target value or the target quantity) is sucked off.

Depending on the variation in the raw coating, the target value for the coating concentration (mean value of the actual coating quantities) is increased to such an extent that all carriers, including those having a minimal loading, will still be within specifications. If, for example, the variation in the raw coating is  $\pm 5\%$ , all carriers will be coated using a mean value of the actual coating quantities of 105 %. This ensures that all parts are coated at  $105 \pm 5\%$  so that all carriers comprise at least the target coating quantity. Hence, the carriers are deliberately overloaded during raw coating. In this exemplary case, the mean value of the actual coating quantities is around 105 % of the required target loading.

Then, the re-suction operation follows. During resuction, the deliberate overload of coating suspension on the carriers is sucked off to the target quantity or close to it.

Any surplus between actual quantity and target quantity resulting from raw coating is preferably determined by weighing. It is particularly preferred to determine the actual quantity on coating suspension by weighing the or each carrier before and after coating and comparing the results. Preferably, the differential quantity which was actually removed is also determined by weighing. If the weight of the carriers designed for coating is sufficiently constant, weighing before coating may be omitted.

If the actual quantity is very close above the target quantity, there will be a risk that re-suction removes so much that the target quantity is not obtained. For that reason, it is advantageous to perform re-suction only if the difference between actual quantity and target quantity exceeds a previously specified threshold value.

The difference between incoming weight of the raw coating and target coating quantity is a criterion for adjusting

the intensity of re-suction. The intensity of re-suction may be adjusted directly through the applied vacuum, or indirectly through an "air damper" or a throttle valve, an air infiltration valve or a calibrated leak in the exhaust line. As another control criterion, the duration of re-suction may be altered. Naturally, both parameters may be changed in a suitable combination in order to adjust the intensity of re-suction.

Preferably, however, a constant time period for re-suction within the range between 0.1 and 5 seconds, in particular between 0.5 and 2 seconds, is used and the intensity of re-suction is adjusted by changing the vacuum using a throttle valve, an air infiltration valve or a calibrated leak. In the simplest case, the intensity of re-suction is adjusted by a controller from previously determined characteristics including a list of the necessary adjustment parameters for the throttle valve etc. depending on the overload, i.e., the difference between incoming weight of the raw coating and target coating quantity.

As a rule, these characteristics depend on the composition of the coating suspension used and, accordingly, must be determined separately for each coating type (for example three-way catalyst for gasoline engines, diesel oxidation catalyst or nitrogen-oxide storage catalyst). Therefore, it may, for example, be useful to provide several air infiltration valves which are adapted in an optimum manner to the control range for various types of coating suspension and/or varying degrees of overload.

Particularly advantageous is the construction of a closed-loop control circuit comprising the carriers to be coated as a controlled system, the measured load as an actual value and the desired load as a target value. Using the deviation between actual values and target values, a controller determines manipulated variables for adjusting the throttle valve (or air infiltration valve etc.) serving as final control element. An increasing campaign duration results in a control function which is refined and improved in a self-learning fashion. Consequently, the adjustment of re-suction

can be carried out for each successive carrier, if process parameters are otherwise constant. Depending on excess coating, the performance of re-suction is individually adjusted in advance for the specific part. The closed-loop control system independently evaluates the success of this action in order to adjust and improve the control parameters.

Thus, the coating of all carriers will be constrained within a specified tolerance window (e.g.  $\pm 1\%$ ) above the target quantity, which would be impossible using a single suction operation.

In particularly preferred embodiments, removal of the surplus or the differential quantity is carried out by iteratively removing a certain, relatively small quantity, weighing and, if necessary, repeating these steps. Thus, steps b) and c) of the method are performed at least twice until the actual quantity is within a previously specified tolerance interval above the target quantity. The specified threshold value may be reduced after each run in order to increase the precision for a renewed run.

During repetition of the above-mentioned steps, the relatively small quantities are preferably sucked from mutually opposite ends of the carrier. This is advantageous in that the uniformity of the coating is improved over the length of the ducts in the carrier. In order to suck the relatively small quantities from mutually opposite ends of the carrier, the carrier is turned 180 degrees each during re-suction, thereby applying its opposite ends to a suction station.

However, iterative re-suction poses the risk that the coating suspension solidifies with increasing iteration stages and that the coating is only increasingly dried by re-suction. This behavior may be compensated for by a corresponding control or closed-loop control program. It is, however, preferred to limit the number of re-suction operations to a maximum of 2 to 3.

After completion of re-suction, the coated carriers are dried at an elevated temperature between 80 and 200 °C for a time period of 5 minutes to 2 hours, and then are usually

calcined at temperatures between 300 and 600 °C for a time period of 10 minutes to 5 hours. Calcination causes the coating to be strongly fixed to the carrier and converts any precursor compounds in the coating suspension into their final form.

The method presented provides excellent accuracy of, i.e. low variation in, the coating concentration when carriers are coated with catalytically active coatings. This increased accuracy is obtained by the re-suction operation according to the invention. Initially, this was surprising as there was a concern that re-suction would only remove the liquid phase of the coating suspension, but not a corresponding solids content, too. However, examinations carried out by the inventors proved that this was not true. The ratio of dry uptake to wet uptake changes only slightly as a result of re-suction.

Therefore, it is possible to shift the mean value of the actual coating quantities achieved with the method so as to be closer to the technically required target coating quantity. As a result, considerable savings of noble metal and valuable raw materials for the coating may be obtained. By contrast, in the conventional coating methods, the mean value of the actual coating quantities must be selected clearly higher than the technically required target coating quantity in order to safely prevent the coating quantity in some carriers from falling below the target value.

Use of this method for the fabrication of multiple layers on the carriers is of particular advantage. Here, the variations in the individual coatings add up, so that with the conventional methods, considerable variations in the final multilayer coating must be taken into account. By applying the method according to the invention to this coating problem, multilayer coatings having clearly reduced variations in the coating concentration may be fabricated.

In the following, preferred exemplary embodiments of the apparatus and the method will be explained in detail with reference to Figures 1 and 2, wherein:

Figure 1 is a schematic representation showing a preferred coating system for performing the method; and

Figure 2 is a diagram showing the coating quantities for a series of carriers when coated conventionally and when coated with the method according to the invention.

Figure 1 shows the possible construction of a coating system suitable for the method. Preferably, the coating system comprises a coating station 20 for producing the raw coating. For this purpose, the carrier 1 to be coated is placed on the retaining elements provided for this purpose, cf. Figure 1. By inflating an inflatable rubber gasket 21, the carrier 1 is fixed and sealed to the station. Further, a second sealing gasket 22 may be provided which is placed on the upper end of the carrier 1 in order to tightly fix an overflow 23. A level sensor 25 is preferably arranged above, which detects a sufficient filling level of the carrier 1 and sends a corresponding signal to the apparatus controller or closed-loop controller of the coating system.

In order to produce the raw coating, the coating suspension is pumped via the feed line 24 into the carrier from below until the level sensor indicates that a specified filling level has been reached. Afterwards, a suction or throttle valve 26 is opened to remove surplus coating suspension from the ducts of the carrier 1 by suction (pre-suction). To do so, a pipe is connected to a vacuum tank (not shown) and a demister. The vacuum tank is connected to a blower which maintains a vacuum, preferably between 50 and 500 and particularly preferably of 300 millibars below ambient pressure. Intensity and duration of pre-suction may be adjusted using the throttle valve 26. They determine the raw coating quantity left on the carrier. In addition, this operation serves to open ducts which may have become blocked by coating suspension.

Figure 1 also shows a weighing station 30 at which the coated carrier 1 is weighed on a balance 31. The quantity of coating suspension in the carrier 1 may be determined in this manner. In addition, a weighing station 10 having a balance

11 may be provided upstream of the coating station 20 so as to determine the weight of the carrier 1 prior to coating.

If it is found in the weighing station 30 that the loading of the carrier 1 with coating suspension is excessively high, the carrier is conveyed onto a re-suction station 40 which removes the excessively applied coating suspension. Similar to the coating station 20, the re-suction station 40 includes a sealing gasket 41 tightly fixing the carrier 1 to the re-suction station 40. A suction valve 46 is used to control the quantity of coating suspension sucked off. If, however, it is found in the weighing station that the applied coating quantity is already below the threshold value, then the carrier is transferred out of the coating system without any re-suction and supplied to a drying and calcining station (not shown).

Following re-suction, it is particularly preferred to weigh the carrier 1 again in the weighing station 30 or another weighing station 50 using a balance 51 as shown in Figure 1. If, during this further check of the quantity of coating suspension in the carrier 1, it is found that there is still too much coating suspension in the carrier 1, the carrier may again be conveyed into the re-suction station 40. Otherwise, the carrier is transferred out of the coating station and supplied to the drying and calcining station.

As indicated before, the weighing stations 30 and 50 may be combined, depending on the desired flexibility and/or speed of the overall system. Moreover, the weighing station 30 and/or 50 may be combined with the re-suction station 40 or the coating station 20.

#### Example

The coating system as shown in Figure 1 was used to perform a coating campaign to coat 500 carriers.

The diagram of Figure 2 shows the coating results for 37 carriers. The coating number is indicated on the abscissa. The ordinate indicates the determined percentage coating quantity based on the desired target quantity (100 %).

Curve a) represents the coating quantities of the raw coating as determined at the weighing station 30. The coating

suspension used had a raw coating variation of  $\pm 3\%$ . Accordingly, the mean value of the raw coating was set to 103 % of the target coating quantity.

Curve b) shows the results after a single re-suction operation. The variation in the coating quantity could be reduced to  $\pm 0.5\%$  by re-suction. The mean value of the coating quantities produced by re-suction was about 101 % of the target quantity.

The coating suspension removed from the carriers by re-suction is collected and re-supplied to the coating process following a corresponding conditioning procedure. As a result, in this example the proposed method can save about 2 % of noble metal cost and corresponding cost for the other coating materials.

**Claims**

1. A method for coating open-pored bodies with at least one coating suspension including, in particular, solids and solutes in a liquid medium, in a quantity in wet state which is to correspond to at least a required target quantity, wherein the coating operation includes a variation in the applied wet coating quantity from one body to the other, the method being characterized by the steps of:
  - (a) coating a body with an actual quantity of the coating suspension, which is always larger than the required target quantity taking the variation of the coating operation into account,
  - (b) determining the difference between the actual quantity and the required target quantity, and
  - (c) reducing the difference between actual quantity and target quantity by removing still wet coating suspension.
2. The method according to claim 1, wherein steps (a) to (c) are followed by drying and calcination of the applied coating suspension.
3. The method according to claim 2, wherein step (c) includes reducing the difference between actual quantity and target quantity by re-suction from one end of the body using an intensity and/or duration matched with the magnitude of the differential quantity.
4. The method according to claim 3, wherein intensity and/or duration of re-suction are selected from tables of values for the measured actual quantity established in preliminary tests.

5. The method according to claim 4, wherein duration and/or intensity of re-suction are controlled in accordance with the values for the actual quantity, duration and/or intensity determined for the bodies coated immediately before, and the associated reduction obtained in the difference between actual and target quantities.
6. The method according to any of the preceding claims, wherein the actual quantity is determined by weighing the body before and after coating.
7. The method according to any of claims 1 to 6, wherein steps (b) and (c) are run at least twice until the actual quantity is within a previously specified tolerance range above the target quantity.
8. The method according to claim 7, wherein re-suction is performed from the second end of the body during the second run.
9. The method according to claim 6, wherein the reduction in the difference between actual quantity and target quantity in step (c) is performed only if said difference exceeds a previously specified threshold value.
10. The method according to claim 7, wherein the reduction in the difference between actual quantity and target quantity in step (c) is performed only if said difference exceeds a previously specified threshold value and said threshold value is reduced after each run.
11. Apparatus for coating open-pored bodies with at least one coating suspension including, in particular, solids and solutes in a liquid medium, in a quantity in wet state which is to correspond to at least a required target quantity, wherein the coating operation includes a variation in the applied wet coating quantity from one body to the other, the apparatus, in particular,

being for a method according to at least one of the preceding claims, comprising:

- (a) a coating station (20) for coating the body with an actual quantity of the coating suspension, which is always larger than the required target quantity taking the variation of the coating operation into account,
- (b) weighing stations (30, 50) for determining the difference between the actual quantity and the required target quantity, and
- (c) a re-suction station (40) for reducing the difference between actual quantity and target quantity by removing still wet coating suspension.

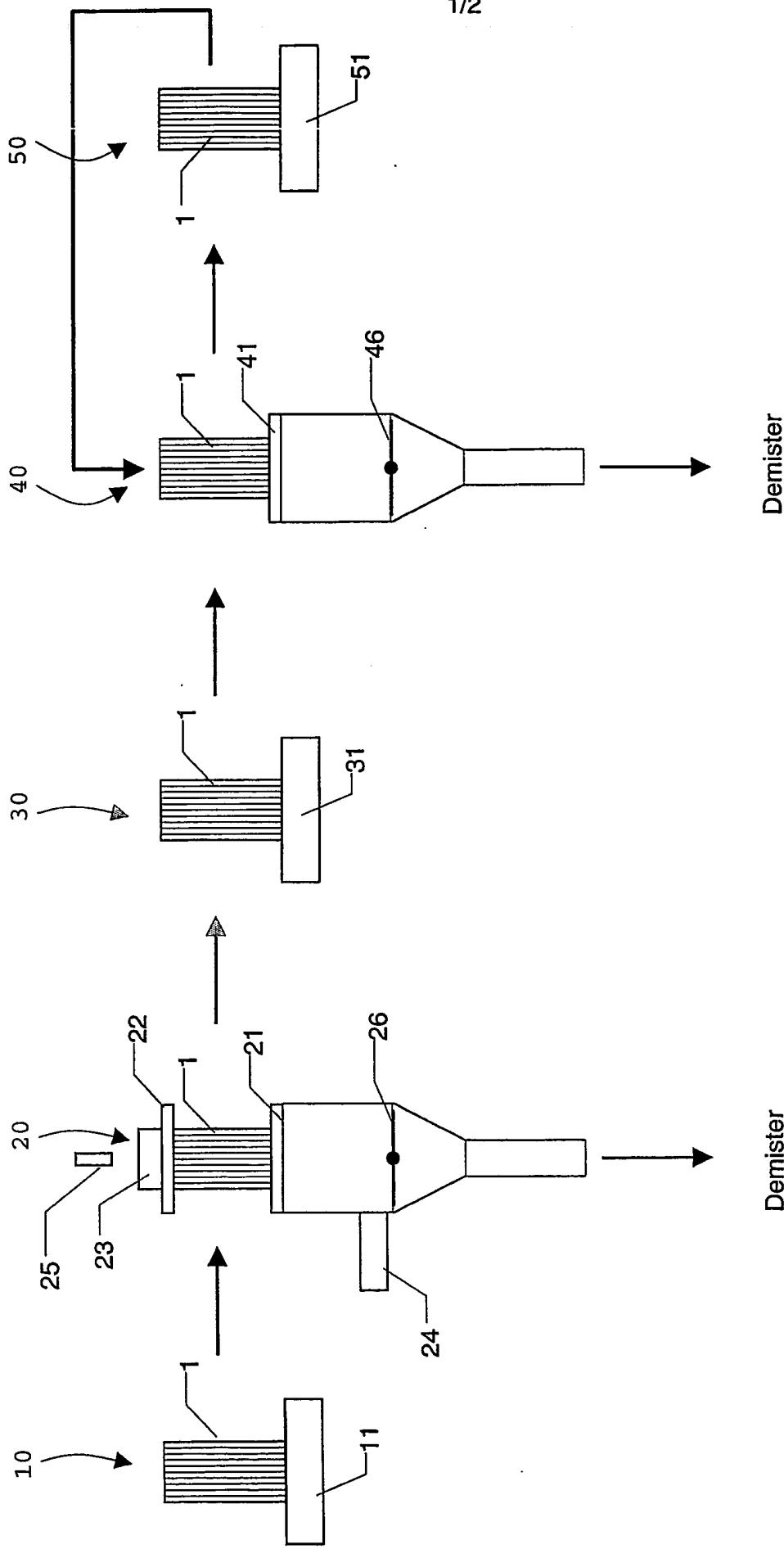
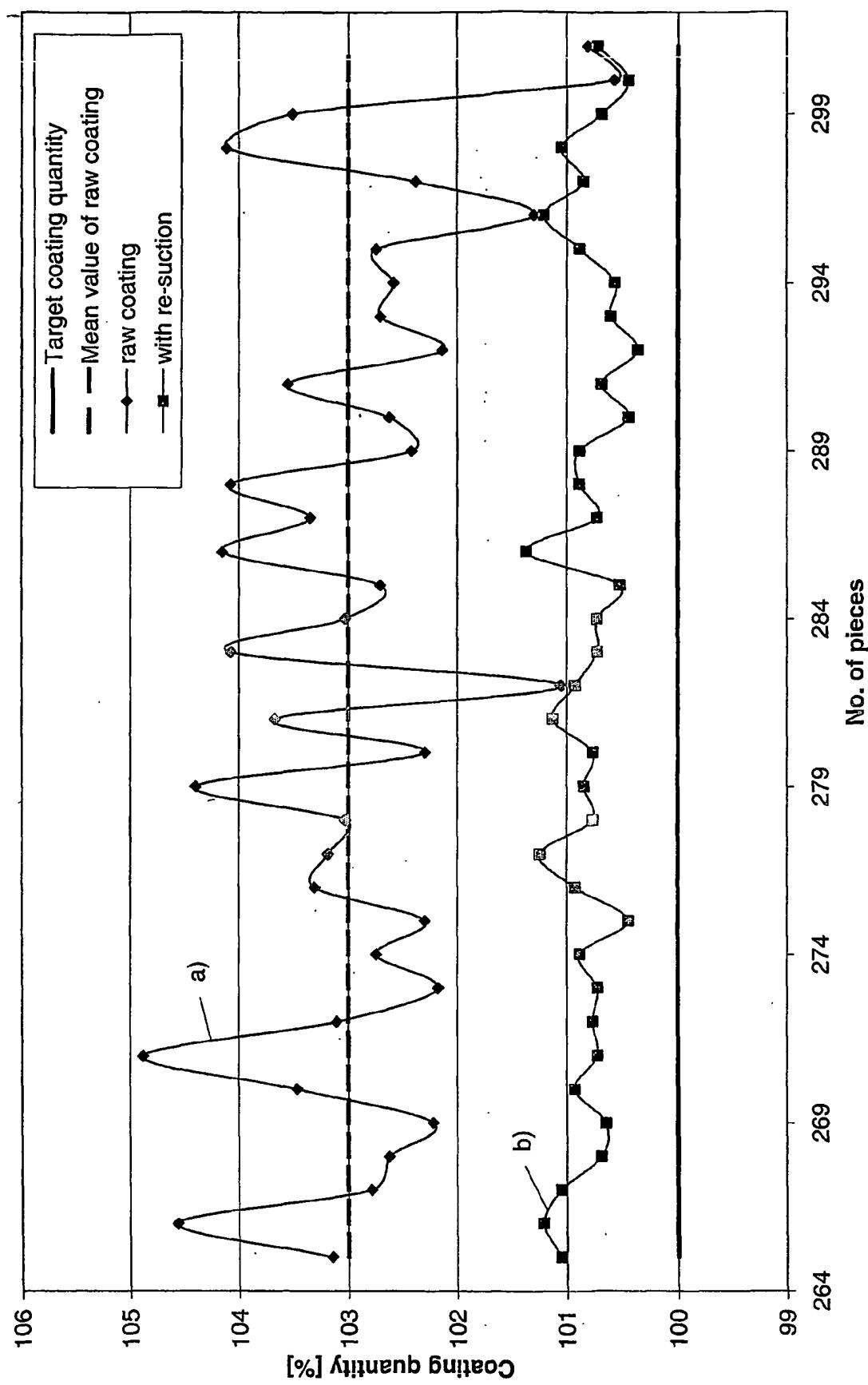


Figure 1

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**Figure 2**

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP2004/004074

**A. CLASSIFICATION OF SUBJECT MATTER**  
**IPC 7 B01J37/02 B01J35/04 B05D7/22**

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
**IPC 7 B01J B05D B05C**

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**EPO-Internal**

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 1 273 344 A (OMG AG & CO KG) 8 January 2003 (2003-01-08) cited in the application paragraphs '0024!, '0030!, '0042! -----	1-11
X	US 3 959 520 A (HOYER WILLIAM A ET AL) 25 May 1976 (1976-05-25) claims 1-4 -----	11
A	US 4 208 454 A (LEAL RICHARD A ET AL) 17 June 1980 (1980-06-17) column 5, line 8 - column 6, line 67; figure 2 -----	1-10
X		11
A		1-10
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

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Date of the actual completion of the International search

13 August 2004

Date of mailing of the International search report

19/08/2004

Name and mailing address of the ISA

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## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP2004/004074

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	KOLB W B ET AL: "THE INS AND OUTS OF COATING MONOLITHIC STRUCTURES. \IMPROVE YOUR UNDERSTANDING OF COATING CATALYTIC SUPPORT MATERIALS" CHEMICAL ENGINEERING PROGRESS, AMERICAN INSTITUTE OF CHEMICAL ENGINEERS. NEW YORK, US, vol. 89, no. 2, 1 February 1993 (1993-02-01), pages 61-67, XP000343928 ISSN: 0360-7275 the whole document -----	1
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